


Identifier: SOP-15.03	Revision: 1	
Effective Date: 05/27/03	Review Date: 04/20/2004	
Document Catalog Number: ER2003-0290		
Author: Karen Schultz-Paige		

Risk Reduction and Environmental Stewardship— Remediation Program

Standard Operating Procedure

for Routine Validation of Organochlorine Pesticides and Polychlorinated Biphenyls Data



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Revision Log

Revision No.	Effective Date	Prepared By	Description of Changes	Affected Pages
R0	04/27/2000	Bart Vanden Plas	Initial procedure.	All
R1	05/27/03	Karen Schultz-Paige	Rewritten to streamline and update process.	All
Review	04/20/2004	Karen Schultz-Paige	Deemed process adequate.	All

Routine Validation of Organochlorine Pesticides and Polychlorinated Biphenyls Data

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Routine Validation of Organochlorine Pesticides and Polychlorinated Biphenyls Data

1.0 PURPOSE

- 1.1 This standard operating procedure (SOP) represents the minimum standards for evaluating routine organochlorine pesticide (PEST) and polychlorinated biphenyl (PCB) analytical data. These data can be generated for the Los Alamos National Laboratory (LANL), Risk Reduction and Environmental Stewardship—Remediation (RRES-R) Program, using SW-846 Method 8081/8082, the comparable Contract Laboratory Program (CLP) methods under the current statement of work (SOW) for analytical services (LANL 1995), or U.S. Environmental Protection Agency (EPA) Method 608 for surface water analyses. The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used to develop focused data validation requirements specific to a particular data use.
- 1.2 Implementation of this procedure results in a tabulation of data compliances and noncompliances identified relative to expectations for data quality based on national guidelines for organic data review U.S. Environmental Protection Agency ([EPA] 1999, 66649). Data noncompliance is noted through the application of qualifiers (Attachment A) and reason codes (Attachment B) to the reported results. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as *general indicators* of data quality and should not be construed as a definitive identification of data usability.
- 1.3 Nothing in this SOP precludes the validator from going beyond the minimum requirements specified in this SOP. In order to address data-quality issues in a data package, the validator may assign qualifiers based on his or her professional judgment. Implementation of this procedure may be followed by a more focused and data-use-specific evaluation of data, especially if implementation of this SOP indicates that the data may contain technical deficiencies. The validator will note any need for a more focused validation on the Data-Validation Cover Sheet (Attachment C). The validator will use the PEST/PCB Data-Validation Checklist (Attachment D) to record the specific validation steps conducted.

2.0 SCOPE

- 2.1 All **RRES-R Personnel** shall implement this mandatory SOP who evaluate routine PEST and PCB analytical data for the RRES-R Program.

- 2.2 **Subcontractors** performing work under the RRES-R Program's quality program shall follow this SOP.

3.0 TRAINING

- 3.1 **RRES-R Personnel** shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.
- 3.2 **RRES-R Personnel** using this SOP shall document training in the RRES-R training database located at <http://erinternal.lanl.gov/Training/login.asp> in accordance with QP-2.2.
- 3.3 The responsible **supervisor** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.
- 3.4 All **data validators** who implement this SOP shall possess a minimum of a bachelors degree in chemistry and two years experience in generating analytical data in an environmental analytical laboratory or two years of data-validation experience.
- 3.5 Inexperienced **validators** shall work under the direct supervision of an experienced, trained RRES-R Program validator. The experienced validator shall review and sign data packages until ten data packages for each analytical suite are satisfactorily validated.
- 3.6 RRES-R Program **validators** shall demonstrate familiarity with the EPA national functional guidelines for organic data review (EPA 1999, 66694).

4.0 DEFINITIONS

- 4.1 *Analyte*—Element, nuclide, or ion (a chemical analysis seeks to identify and/or quantify the chemical constituent of interest).
- 4.2 *Area count*—Integrated area under a chromatographic peak. The area count is proportional to the amount of compound present in the aliquot introduced into the chromatograph.
- 4.3 *Continuing calibration verification (CCV)*—Check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A CCV is performed every 12 h of operation or (for inorganic and high explosives [HEs]) every ten injections (samples and/or quality control [QC] samples), whichever is more frequent, thus verifying the satisfactory performance of an instrument on a day-to-day basis. The continuing calibration 12-h period assumes that the instrument has not been shut down since the initial calibration.

- 4.4 *Data validator*—Person who has met the minimum standards of training established by the RRES-R Program for data validation and who performs data validation on behalf of the RRES-R Program (hereinafter referred to as the “validator”).
- 4.5 *Detect (inorganic and organic)*—Sample result above the method detection limit (MDL) reported by the contract analytical laboratory. The contract laboratory reports the concentration of the analyte in the sample.
- 4.6 *Form 1*—Organic analysis data sheet for each individual sample that includes the sample information needed to identify the sample and the analytical results for the sample. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.
- 4.7 *Holding time*—The maximum elapse of time that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions, and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.
- 4.8 *Initial calibration*—Process used to establish the relationship between instrument response and analyte concentration at several analyte-concentration values to demonstrate that an instrument is capable of acceptable analytical performance.
- 4.9 *Instrument performance check*—Analysis of a chemical of known relative mass abundances that indicates how well a mass spectrometer is calibrated.
- 4.10 *Internal standard (IS)*—Chemical compound added to every blank, sample, and standard extract at a known concentration that is used to (1) compensate for analyte concentration changes that might occur during storage of the extract and (2) compensate for quantitation variations that can occur during analysis. ISs are used as the basis for quantitation of target analytes.
- 4.11 *Laboratory control sample (LCS)*—Known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.
- 4.12 *Laboratory duplicate sample*—Portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is equally representative of the original material. Duplicate analyses also are

performed to generate data, and to determine the long-term precision of an analytical method on various matrices.

- 4.13 *Laboratory qualifier (or laboratory flag)*—Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines (EPA 1994, 48639; EPA 1999, 66649).
- 4.14 *LANL data-validation qualifiers*—Data qualifiers defined by LANL and used in the RRES-R Program routine-validation process. Attachment A lists all data qualifiers that are applicable to all analytical suites.
- 4.15 *LANL data-validation reason codes*—Codes applied to the sample data by data validators who are independent of the contract laboratory that performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier along with a description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate RRES-R Program SOP.
- 4.16 *Lower acceptance limit (LAL)*—Lowest limit that is acceptable, based on the quality control (QC) criteria for a specific QC sample for a specific method. Any results lower than the LAL are qualified following this routine validation procedure.
- 4.17 *Matrix spike*—An aliquot of sample spiked with a known concentration of target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. Spiking typically occurs before sample preparation and analysis.
- 4.18 *Method blank*—Analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. A method blank is used to assess the potential for sample contamination during preparation and analysis.
- 4.19 *Method detection limit (MDL)*—Minimum concentration of a substance that can be measured and reported with known statistical confidence that the analyte concentration is greater than zero. The MDL is determined by analysis of samples of a given matrix type that contain the analyte after the sample is subjected to the usual preparation and analyses. The MDL is used to establish detection status.
- 4.20 *Nondetect (organics)*—Sample result that is less than the MDL. The laboratory reports nondetects as undetected at the reporting limit (RL).
- 4.21 *Percent difference (%D)*—Measure of deviation from the initial calibration to the continuing calibration, based on calibration factors.

- 4.22 *Percent recovery (%R)*—Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.
- 4.23 *Percent relative standard deviation (%RSD)*—Evaluation of deviation between the concentrations versus analyte response over the dynamic linear calibration range. The basic equation is $\%RSD = (\text{Std dev}/\text{av}) \bullet 100$.
- 4.24 *Relative response factor (RRF)*—Relationship between analyte concentrations versus area response.
- 4.25 *Reporting limit (RL)*—Lowest concentration that reliably can be achieved within specified limits of precision and accuracy during routine analytical-laboratory operating conditions. The low point on a calibration curve should reflect this reporting limit. The RL is not used to establish detection status.
- 4.26 *Request number (RN)*—An identifying number assigned by the RRES-R Program to a group of samples that are submitted for analysis.
- 4.27 *Routine data*—Data generated using analytical methods that are identified as routine methods in the current RRES-R Program SOW for analytical services.
- 4.28 *Routine Data-Validation*—Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is two-fold: (1) to estimate the technical quality of the data relative to minimum national guidelines adopted by the RRES-R Program; (2) to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.
- 4.29 *Surrogate compound (surrogate)*—Organic chemical compound used in the analyses of organic target analytes that is similar in composition and behavior to target analytes but is not normally found in environmental samples. Surrogates are added to every blank, sample, and spike to evaluate the efficiency with which analytes are recovered during extraction and analysis.
- 4.30 *Target analyte*—An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.
- 4.31 *Tentatively identified compound (TIC)*—Chemical compound detected in a sample that is not a target analyte, IS, or surrogate compound. Up to 30 chromatographic peaks may be subject to mass spectral matching for identification as TICs. Tentative identification is based on comparison of the compound mass spectrum to an industry-standard mass-spectra library

using both a statistical matching algorithm and the professional judgment of the analyst.

- 4.32 *Upper acceptance limit (UAL)*—Highest limit that is acceptable, based on the QC criteria for a specific QC sample for a specific method. All results greater than the UAL are qualified following this routine validation procedure.

5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure:

- RRES-R Personnel
- Project Team Leader
- Quality Program Project Leader
- Supervisor
- User

6.0 PROCEDURE

Data Validators shall perform the following processes.

6.1 Preparing for Data Validation

1. Obtain the required current version of the PEST/PCB Data-Validation Checklist form (Attachment D) from the RRES-R Program website (<http://erinternal.lanl.gov/quality/forms.htm>).
2. Obtain from the Sample Management Office (SMO) of the Field Support Facility (FSF) all data packages that contain the sample data to be validated.
 - A. Prepare a PEST/PCB Data-Validation Cover Sheet (Attachment C) by completing the top part of the cover sheet and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.
 - B. If any data are rejected, check the rejected box and notify the project chemist immediately.

Note: The validator may use a single cover sheet or multiple cover sheets when validating multiple analytical suites under the same RN.

Note: Some of the steps in this procedure apply only to the PEST or PCB analyses. In cases where only one type of analysis is being validated, check “n/a” (not analyzed) on the PEST/PCB

Data-Validation Checklist for those steps that do not apply to the data being validated.

Note: Use a separate sheet of paper to document each deficiency identified beyond the scope of this procedure, including phone conversations with the analytical laboratory concerning these deficiencies. Attach these sheets to the PEST/PCB Data-Validation Cover Sheet.

3. Verify that the following items are present in the data package:

- The signed LANL COC record
- The case narrative
- The result forms (CLP Form 1 or equivalent) for each sample
- The QC forms (CLP Form II, III, IV, VII, VIII, IX, X, or equivalent) for water and/or soils, as appropriate
- The chromatograms, quantitation reports, and confirmation data for all samples and blanks.

4.	IF the data-package documentation is...	AND...	THEN...
	Present,		<ul style="list-style-type: none"> • Go to Step 6.
	Missing,	< 6 mo.,	<ul style="list-style-type: none"> • Contact the analytical laboratory. • Allow 3 business days for submittal. • Go to Step 5.
	Missing,	= 6 mo.,	<ul style="list-style-type: none"> • Contact the analytical laboratory. • Allow 10 business days for submittal. • Go to Step 5.

Note: To expedite the validation process, the validator may request that the contract laboratory forward the missing information by e-mail or fax directly to the validator within 24 h of notification.

5.	IF the analytical laboratory...	THEN...
	Submits the documentation	<ul style="list-style-type: none"> • Go to Step 6.

within the specified time period,	
Does <u>not</u> submit documentation within the specified time period,	<ul style="list-style-type: none"> • Notify the SMO for contract-compliance action. • Go to Step 6.

6. Record the presence or absence (“Yes” or “No” or “n/a”) of each item, as appropriate, in the Completeness Check Section of the Data-Validation Cover Sheet.

Note: Indicate any samples whose data are missing from the data package under comments/problems noted.

7. Photocopy the following:

- The chain of custody forms
- The Form 1 that will be used during the validation process

Note: Do not record data-validation qualifiers and reason codes on the original Form 1.

Note: Each page of the Form 1 must be initialed and dated by the validator; this must be done even if the validator accepts the analytical laboratory qualification.

8. Submit photocopies of the items listed in Step 7 as attachments to the completed PEST/PCB Data-Validation checklists.

9. Go to Section 6.2, “Verifying the Analyte Breakdown.”

6.2 Verifying the Analyte Breakdown

Note: Use Table 6.2-1 to identify primary analytes and associated breakdown products.

Table 6.2-1
Breakdown Analytes

Primary Breakdown Analyte	Associated Breakdown Products
4,4'-DDT	DDD & DDE
Endrin	Endrin aldehyde & Endrin ketone

1. IF the sample analyzed was for...	THEN...
--------------------------------------	---------

PCBs <u>only</u> ,	<ul style="list-style-type: none"> • Check “n/a” on lines 1,2, and 3 of the PEST/PCB Data-Validation Checklist (Attachment D). • Go to Section 6.3, “Verifying the Initial Calibration.”
PCBs and something else,	<ul style="list-style-type: none"> • Go to Step 2.

2. IF the breakdown information is...	THEN...
Present,	<ul style="list-style-type: none"> • Go to Step 3.
Required but missing,	<ul style="list-style-type: none"> • Check “Yes” on line 1 and “n/a” on lines 2 and 3 of the PEST/PCB Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory cannot provide the breakdown information, qualify all the 4,4'-DDT, DDD, DDE, Endrin, Endrin aldehyde and Endrin ketone results as rejected (R, P10b). • Go to Section 6.3, “Verifying the Initial Calibration.”

3. IF the breakdown results...	THEN...
Passed the acceptance criteria,	<ul style="list-style-type: none"> • Check “No” on lines 1, 2, and 3 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.3, “Verifying the Initial Calibration.”
Did <u>not</u> pass the criteria,	<ul style="list-style-type: none"> • Go to Step 4.

4. If the breakdown is greater than 20% for either DDT or Endrin or if the combined breakdown is less than 30%,
- A. Check “No” on line 1 and “Yes” on line 2 of the PEST/PCB Data-Validation Checklist.

B. Determine if DDT or Endrin are present in the sample.

C.	IF, in the sample...	THEN...
	<u>Either</u> DDT or Endrin are present,	<ul style="list-style-type: none"> • Qualify the primary analyte present as estimated with a potential negative bias (J-, P10) on the individual sample Form 1. • Determine if the breakdown products of this primary analyte are present in the sample. • Go to Step 5.
	<u>Neither</u> DDT or Endrin are present,	<ul style="list-style-type: none"> • Go to Step 6.

5.	IF the primary breakdown analyte and the associate products are...	THEN...
	<u>Not</u> present in the sample,	<ul style="list-style-type: none"> • Go to Step 6.
	Present in the sample,	<ul style="list-style-type: none"> • Check "Yes" on line 3 of the PEST/PCB Data-Validation Checklist. • Qualify the breakdown products as estimated with a potential positive bias (J+, P10a) on the individual sample Form 1. • Go to Step 6.

6.	IF either primary breakdown analyte is...	THEN...
	Detected in the sample,	<ul style="list-style-type: none"> • Go to Step 7.
	<u>Not</u> detected in the sample,	<ul style="list-style-type: none"> • Determine if breakdown products are present in the sample. • If the breakdown products are present, go to Step 7. <p>OR</p> <ul style="list-style-type: none"> • If the breakdown products are <u>not</u> present in the sample, go to Step

6.	IF either primary breakdown analyte is...	THEN...
		8.

7.	IF the breakdown products of the undetected primary analytes are...	THEN...
	<u>Not</u> present in the sample,	<ul style="list-style-type: none"> Go to Step 8.
	Present in the sample,	<ul style="list-style-type: none"> Check "Yes" on line 3 of the PEST/PCB Data-Validation checklist. Qualify the primary analyte results as rejected (R, P10) on the individual sample Form 1. Qualify the breakdown product results as estimated with a potential positive bias (J+, P10a) on the individual sample Form 1. Go to Section 6.3, "Verifying the Initial Calibration."

8.	IF either primary breakdown analyte is...	THEN...
	Detected in the sample, <u>and</u> if the associated breakdown products are present in the sample,	<ul style="list-style-type: none"> Go to Section 6.3, "Verifying the Initial Calibration."
	<u>Not</u> detected in the sample, <u>and</u> if the associated breakdown products are <u>not</u> present in the sample,	<ul style="list-style-type: none"> Check "No" on line 3 of the PEST/PCB Data-Validation Checklist. Qualify the primary analyte as estimated (UJ, P10) on the individual sample Form 1. Qualify the breakdown products as estimated (UJ, P10a) on the individual sample Form 1. Go to Section 6.3, "Verifying the Initial Calibration."

6.3 Verifying the Initial Calibration

1. IF the initial calibration information is...	THEN...
Present,	<ul style="list-style-type: none"> Go to Step 2.
Missing,	<ul style="list-style-type: none"> Check "Yes" in line 4 and "n/a" on lines 5 and 6 of the PEST/PCB Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, P16) on the individual sample Form 1. Go to Step 2.

2. IF the initial calibration...	THEN...
Has five calibration points,	<ul style="list-style-type: none"> Check "No" on lines 4 and 5 of the PEST/PCB Data-Validation Checklist. Go to Step 3.
Does <u>not</u> have five calibration points,	<ul style="list-style-type: none"> Check "No" on line 4 and "Yes" on line 5 of the PEST/PCB Data-Validation Checklist. Qualify the detected analytes as estimated (J, P7) and the undetected analytes as rejected (R, P7) on the individual sample Form 1. Go to Step 3.

3. IF the percent relative standard deviation (%RSD) for...	THEN...
<u>Each</u> analyte is = 20% (If the %RSD is not used, then the correlation coefficient must be = 0.995),	<ul style="list-style-type: none"> Check "No" on line 6 of the PEST/PCB Data-Validation Checklist. Go to Section 6.4, "Verifying the

	Continuing Calibration.”
<u>Any</u> analyte is > 20% (or if the correlation coefficient is < 0.995),	<ul style="list-style-type: none"> • Check “Yes” on line 6 of the PEST/PCB Data-Validation Checklist. • Qualify all the the affected analytes as estimated (J, P7a/UJ, P7a) on the individual sample Form 1. • Go to Section 6.4, “Verifying the Continuing Calibration.”

6.4 Verifying the Continuing Calibration

1. IF the continuing calibration information is...	THEN...
Present,	<ul style="list-style-type: none"> • Go to Step 2.
Missing <u>or</u> not analyzed at the proper frequency,	<ul style="list-style-type: none"> • Check “Yes” on line 7 and “n/a” on line 8 of the PEST/PCB Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, P16) on the individual sample Form 1. • Go to Step 2.

2. IF the percent difference (%D) for...	THEN...
<u>Each</u> analyte is = 15%,	<ul style="list-style-type: none"> • Check “No” on lines 7 and 8 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.5, “Verifying the Multicomponent Analyte Continuing Calibration.”
<u>Any</u> analyte is > 15%,	<ul style="list-style-type: none"> • Check “No” on line 7 and “Yes” on line 8 of the PEST/PCB Data-Validation Checklist. • Qualify all the affected analytes as estimated (J, P7a/UJ, P7a) on the individual sample Form 1. <p>Note: The qualification of the data for any continuing calibration problem affects the samples injected before and after the failing CCV. The validator must qualify all the samples that are affected.</p> <ul style="list-style-type: none"> • Go to Section 6.5, “Verifying the Multicomponent Analyte Continuing Calibration.”

6.5 Verifying the Multicomponent Analyte Continuing Calibration

Note: This validation check is required for all multicomponent analyte detections because the contract analytical laboratory must analyze the calibration standard within 72 h after the detection of any multicomponent analyte. Furthermore, verify that there was a standard of the multicomponent analyzed during the initial calibration and that the standard passes CCV criteria.

IF a standard of the multicomponent analyte was...	THEN...
<u>Not</u> required,	<ul style="list-style-type: none"> • Check “n/a” on line 9 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.6, “Verifying the Retention Times.”
Analyzed within 72 h after the detection of a multicomponent	<ul style="list-style-type: none"> • Check “No” on line 9 of the PEST/PCB Data-Validation

analyte in a sample,	<p>Checklist.</p> <ul style="list-style-type: none"> Go to Section 6.6, "Verifying the Retention Times."
<u>Not</u> analyzed within 72 h after the detection of a multicomponent analyte in a sample, <u>and/or</u> was not analyzed during the initial calibration, <u>and/or</u> does not pass acceptance criteria,	<ul style="list-style-type: none"> Check "Yes" on line 9 of the PEST/PCB Data-Validation Checklist. Qualify all the affected detected analytes as estimated (J, P7b) on the individual sample Form 1. Go to Section 6.6, "Verifying the Retention Times."

6.6 Verifying the Retention Times

1.	IF the retention times window information is,	THEN...
	Present,	<ul style="list-style-type: none"> Go to Step 2.
	Missing,	<ul style="list-style-type: none"> Check "Yes" in line 10 and "n/a" on line 11 of the PEST/PCB Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, P11) on the individual sample Form 1. Go to Step 2.
2.	IF the retention times for any of the compounds have...	THEN...
	<u>Not</u> shifted by more than 0.05 min from the initial calibration,	<ul style="list-style-type: none"> Check "No" on lines 10 and 11 of the PEST/PCB Data-Validation Checklist. Go to Section 6.7, "Verifying the Method-Blank Results."

2.	IF the retention times for any of the compounds have...	THEN...
	Shifted by more than 0.05 min from the initial calibration,	<ul style="list-style-type: none"> • Check “No” on line 10 and “Yes” on line 11 of the PEST/PCB Data-Validation Checklist. • Using professional judgment, reject (R, P11a) any anomalous results where the detect status can’t be clearly determined based on the available retention time information. • Qualify all the results outside the initial calibration retention time window as undetected but estimated (UJ, P11a) on the individual sample Form 1. • Qualify all the results inside the initial calibration retention time window as detected but estimated (J, P11a) on the individual sample Form 1. • Go to Section 6.7, “Verifying the Method-Blank Results.”

6.7 Verifying the Method-Blank Results

Note: The data validator must compare method-blank results to the contractually required estimated quantitation limits (EQLs).

1.	IF the method-blank information is...	THEN...
	Present,	<ul style="list-style-type: none"> • Go to Step 2.

1.	IF the method-blank information is...	THEN...
	Missing,	<ul style="list-style-type: none"> • Check “Yes” in line 12 and “n/a” on lines 13 and 14 of the PEST/PCB Data-Validation Checklist. • Contact the analytical laboratory and SMO to request the missing information (see Section 6.1 -4). • If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, P4b) on the individual sample Form 1. • Go to Step 2.

2.	IF the method blank has...	THEN...
	Detected results,	<ul style="list-style-type: none"> • Go to Step 3.
	<u>No</u> detected results,	<ul style="list-style-type: none"> • Check “No” on lines 12, 13, and 14 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.8, “Verifying the Confirmation Results.”

3.	IF the concentration of any analyte in a sample is...	THEN...
	= 5 times the concentration of that analyte detected in the corresponding blank,	<ul style="list-style-type: none"> • Check “No” on line 12 and “Yes” on line 13 of the PEST/PCB Data-Validation Checklist. • Qualify all the affected analytes as undetected but estimated (UJ, P4) on the individual sample Form 1. • Go to Section 6.8, “Verifying the Confirmation Results.”

3.	IF the concentration of any analyte in a sample is...	THEN...
	> 5 times the concentration of that analyte detected in the corresponding blank,	<ul style="list-style-type: none"> • Check “No” on line 12 and “Yes” on line 14 of the PEST/PCB Data-Validation Checklist. • Qualify all the affected analytes as estimated (J, P4a) on the individual sample Form 1. • Go to Section 6.8, “Verifying the Confirmation Results.”

Note: Check concentrations of common laboratory contaminants in the diluted samples. If the concentration is within ten times the concentration of the blank minus the dilution factor and if the sample was NOT diluted for that analyte, use professional judgment to apply the qualification criteria listed in this section.

6.8 Verifying the Confirmation Results

Note: Only detected results need confirmation. The confirmation column is held to the same criteria as the primary column for all qualification criteria.

1.	IF the confirmation information is...	THEN...
	Present,	<ul style="list-style-type: none"> • Go to Step 2.
	Required but missing,	<ul style="list-style-type: none"> • Check “Yes” on line 15 and “n/a” on lines 16, 17 and 18 of the PEST/PCB Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all the affected results as rejected (R, P8a) on the individual sample Form 1. • Go to Step 2.

2.	IF the analyte is detected and...	BUT...	THEN...
	<u>Not</u> confirmed,		<ul style="list-style-type: none"> • Check “No” online 15, “Yes” on line 16, and “n/a” on lines 17 and 18 of the PEST/PCB Data-Validation Checklist. • Qualify all the affected analytes as undetected (UJ, P8) on the individual Form 1. • Go to Section 6.9, “Verifying the Surrogate Recoveries.”
	Confirmed with adequate pattern recognition, and confirmation passes all QC requirements,		<ul style="list-style-type: none"> • Check “No” on lines 15,16, 17 and 18 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.9, “Verifying the Surrogate Recoveries.”
	Confirmed,	Outside the acceptable RT windows listed in the data package by the analytical laboratory,	<ul style="list-style-type: none"> • Check “No” on lines 15,16 and 18 and “Yes” on line 17 of the PEST/PCB Data-Validation Checklist. • Qualify all the affected analytes as estimated (J, P11b) on the individual Form 1. • Go to Section 6.9, “Verifying the Surrogate Recoveries.”
	Confirmed,	Does not resemble the pattern from the standard,	<ul style="list-style-type: none"> • Check “Yes” on line 18 of the PEST/PCB Data-Validation Checklist. • Qualify all the affected analytes as estimated (J, P7b) on the individual Form 1. • Go to Section 6.9, “Verifying the Surrogate Recoveries.”

6.9 Verifying the Surrogate Recoveries

Note: Surrogate percent recovery (%R) values that are out of specification as a result of sample dilution used to bring the detected analyte concentrations into the instrument calibration range are not subject to the acceptance criteria listed in Table 6.9-1.

Table 6.9-1
PEST/PCB Surrogates and Recovery Acceptance Ranges

Surrogate	Soil Matrix Acceptance Range (%R)	Water Matrix Acceptance Range (%R)
Tetrachloro-m-xylene (required for all suites)	60–150	60–150
Decachlorobiphenyl (required for all suites)	60–150	60–150

1.	IF the surrogate information is...	THEN...
	Present,	<ul style="list-style-type: none"> Go to Step 2.
	Missing,	<ul style="list-style-type: none"> Record “Yes” on line 19 of the PEST/PCB Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify all the affected results as rejected (R, P3f) on the individual sample Form 1. Go to Step 2.
2.	IF the surrogate %R in a sample is...	THEN...
	= the upper acceptance limit (UAL),	<ul style="list-style-type: none"> Record “No” on lines 19 and 20 of the PEST/PCB Data-Validation Checklist. Go to Step 3.

2. IF the surrogate %R in a sample is...	THEN...
> the UAL,	<ul style="list-style-type: none"> Record "Yes" on line 20 and "No" on line 19 of the PEST/PCB Data-Validation Checklist. Qualify all the positive analytes as estimated with a potential positive bias (J+, P3) on the individual sample Form 1. Go to Step 3.
3. IF the surrogate %R in a sample is...	THEN...
= the LAL,	<ul style="list-style-type: none"> Record "No" on line 21 of the PEST/PCB Data-Validation Checklist. Go to Section 6.10, "Verifying the Laboratory Control Sample Recoveries."
< the LAL but = 10%,	<ul style="list-style-type: none"> Record "Yes" on line 21 of the PEST/PCB Data-Validation Checklist. Qualify the detected analytes as estimated with a potential negative bias (J-, P3a) and nondetected analytes as estimated (UJ, P3c) on the individual sample Form 1. Go to Step 4.
4. IF...	THEN...
<u>No</u> surrogate %R in a sample is < 10%,	<ul style="list-style-type: none"> Record "No" on line 22 of the PEST/PCB Data-Validation Checklist. Go to Step 5.

4.	IF...	THEN...
	<u>Any</u> surrogate %R in a sample is < 10%,	<ul style="list-style-type: none"> Record "Yes" on line 22 of the PEST/PCB Data-Validation Checklist. Qualify the detected analytes as estimated with a potential negative bias (J-, P3b) and nondetected analytes as rejected (R, P3d) on the individual sample Form 1. Go to Step 5.

5.	IF...	THEN...
	<u>No</u> surrogate %R in a sample is < LAL <u>or</u> no surrogate %R is > UAL,	<ul style="list-style-type: none"> Record "No" on line 23 of the PEST/PCB Data-Validation Checklist. Go to Section 6.10, "Verifying the Laboratory Control Sample Recoveries."
	<u>One</u> surrogate %R in a sample is < LAL <u>and</u> any other surrogate %R is > UAL,	<ul style="list-style-type: none"> Record "Yes" on line 23 of the PEST/PCB Data-Validation Checklist. Qualify all the affected analytes as estimated (J, P3e/UJ, P3e) on the individual sample Form 1. Go to Section 6.10, "Verifying the Laboratory Control Sample Recoveries."

6.10 Verifying the Laboratory Control Sample Recoveries

Note: The analytical laboratory may perform either a full analyte laboratory control sample (LCS) or a short spike list (CLP matrix spike list) (EPA 1999, 66649).

1.	IF the LCS Information is...	THEN...
	Present,	<ul style="list-style-type: none"> Go to Step 2.

1.	IF the LCS Information is...	THEN...
	Missing,	<ul style="list-style-type: none"> • Check “Yes” on line 24 and “n/a” on lines 25, 26 and 27 of the PEST/PCB Data-Validation Checklist. • Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). • If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, P12) on the individual sample Form 1. • Go to Step 2.

2.	IF...	THEN...
	<u>No</u> LCS analyte %R is > 130%,	<ul style="list-style-type: none"> • Check “No” on lines 24 and 25 of the PEST/PCB Data-Validation Checklist. • Go to Step 3.
	<u>Any</u> LCS analyte %R is > 130%,	<ul style="list-style-type: none"> • Check “Yes” on line 25 and “No” on line 24 of the PEST/PCB Data-Validation Checklist. • Qualify all the detected analytes as estimated with a potential positive bias (J+, P12d) on the individual sample Form 1. • Go to Step 3.

3.	IF...	THEN...
	<u>No</u> LCS analyte %R is < 70%,	<ul style="list-style-type: none"> • Check “No” on lines 26 and 27 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.11, “Verifying Holding-Time Results.”

3.	IF...	THEN...
	Any LCS analyte %R is < 70% but = 10%,	<ul style="list-style-type: none"> Check "Yes" on line 26 and "No" on line 27 of the PEST/PCB Data-Validation Checklist. Qualify all the detected analytes as estimated with a potential negative bias (J-, P12b) and undetected analytes as estimated (UJ, P12c) on the individual sample Form 1. Go to Section 6.11, "Verifying Holding-Time Results."
	Any LCS analyte %R is < 10%,	<ul style="list-style-type: none"> Check "Yes" on line 27 and "No" on line 26 of the PEST/PCB Data-Validation Checklist. Qualify all the detected analytes as estimated with a potential low bias (J-, P12a) and undetected analytes as rejected (R, P12a) on the individual sample Form 1. Go to Section 6.11, "Verifying Holding-Time Results."

6.11 Verifying Holding-Time Results

Table 6.11-1
Holding-Time Acceptance Criteria

Sample Matrix	Extraction Holding Time (days)	Analysis Holding Time (days)
Soil	14	40
Water	7	40
The current SOW for analytical services lists applicable storage conditions.		

1.	IF...	THEN...
	All the samples were extracted and analyzed within their holding times,	<ul style="list-style-type: none"> Check "No" on lines 28, 29, and 30 of the PEST/PCB Data-Validation Checklist. Go to Section 6.12, "Verifying the Dilutions."

1.	IF...	THEN...
	Any samples were <u>not</u> extracted within the holding-time acceptance criteria,	<ul style="list-style-type: none"> • Check “Yes” on line 28 of the PEST/PCB Data-Validation Checklist. • Calculate the number of days by which the holding time was exceeded. • Go to Step 2.
2.	IF the extraction holding time did...	THEN...
	<u>Not</u> exceed 2 times the holding-time acceptance criteria,	<ul style="list-style-type: none"> • Check “No” on line 29 of the PEST/PCB Data-Validation Checklist. • Qualify all the detected analytes as estimated with potential negative bias (J-, P9) and undetected analytes as estimated (UJ, P9) on the individual sample Form 1. • Go to Step 3.
	Exceed 2 times the holding-time acceptance criteria,	<ul style="list-style-type: none"> • Check “Yes” on line 29 on the Data-Validation Checklist. • Qualify all the affected results as rejected (R, P9a) on the individual sample Form 1. • Go to Step 3.
3.	IF the analytical holding time was...	THEN...
	<u>Not</u> exceeded,	<ul style="list-style-type: none"> • Check “No” on line 30 of the PEST/PCB Data-Validation Checklist. • Go to Section 6.12, “Verifying the Dilutions.”
	Exceeded,	<ul style="list-style-type: none"> • Check “Yes” on line 30 on the Data-Validation Checklist. • Qualify all the affected results as rejected (R, P9b) on the individual sample Form 1. • Go to Section 6.12, “Verifying the Dilutions.”

6.12 Verifying the Dilutions

IF the sample was...	THEN...
<u>Not</u> diluted,	<ul style="list-style-type: none"> Record "No" on line 31 of the PEST/PCB Data-Validation Checklist. Go to Section 6.13, "Identifying the Obvious Quality Deficiencies."
Diluted <u>and</u> there are no target analytes detected above the second lowest standard,	<ul style="list-style-type: none"> Record "Yes" on line 31 of the PEST/PCB Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide proof of a matrix interference that can't be removed by acceptable cleanup techniques, qualify affected samples (R, P10c). <p>OR</p> <ul style="list-style-type: none"> If the laboratory can provide proof of matrix interference that was not removed by acceptable cleanup attempts, qualify all the nondetected analytes (UJ, P10c) on the individual sample Form 1. Go to Section 6.13, "Identifying the Obvious Quality Deficiencies."

6.13 Identifying the Obvious Quality Deficiencies

1. IF there are...	THEN...
<u>No</u> obvious data quality deficiencies (other than those covered by this SOP),	<ul style="list-style-type: none"> Record "No" on line 32 of the PEST/PCB Data-Validation Checklist. Go to Section 6.14, "Completing the Data-Validation Cover Sheet."

1.	IF there are...	THEN...
	Any obvious/significant data quality deficiencies noted during the data-validation process,	<ul style="list-style-type: none"> • Record "Yes" on line 32 of the PEST/PCB Data-Validation Checklist. • Contact the analytical laboratory and the SMO, if necessary, to resolve the quality issue. • Record the appropriate qualifier to the data based on the validator's best professional judgment and applies Reason Code R19. • Write up a clear description of the quality issue that you flagged on the Data-Validation Cover Sheet. • Go to Section 6.14, "Completing the Data-Validation Cover Sheet."

2. Ensure signature/date.
3. Go to Section 6.14, "Assembling and Submitting the Validation Data-Record Package."

6.14 Assembling and Submitting the Validation Data-Record Package

1. Assemble the following items in order:
 - The completed Data-Validation Cover Sheet
 - PEST/PBC Data-Validation Checklists, Sections 6.2 through 6.11 completed
 - Photocopies of completed Form 1s (containing validator qualifier flags and reason codes)
 - A photocopy of the data-package case narrative
 - Chain of custody forms
2. Submit the Validation-Data Record Package to the FSF in accordance with SOP-15.09, "Chain of Custody for Analytical Data Packages."
3. Go to Section 6.16, "Lessons Learned."

7.0 LESSONS LEARNED

- 7.1 Before performing work described in this SOP, RRES-R Personnel should go to the Department of Energy Lessons Learned Information Services

home page, located at <http://www.tis.eh.doe.gov/II/II.html>, and/or to the LANL Lessons Learned Resources web page, located at http://www.lanl.gov/projects/lessons_learned/, and search for applicable lessons.

- 7.2 During work performance and/or after the completion of work activities, RRES-R Personnel, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at http://www.lanl.gov/projects/lessons_learned/.

8.0 RECORDS

Although no records are submitted to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.13 are part of the data record package submitted to the RPF in accordance with SOP-15.09.

9.0 REFERENCES

To properly implement this SOP, **RRES-R Personnel** should become familiar with the contents of the following documents located at http://erinternal.lanl.gov/home_links/Library_proc.shtml:

- EPA (US Environmental Protection Agency), "US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," Publication 9240.1-05, EPA-540/R-94/012, Office of Solid Waste and Emergency Response, Washington, DC (February 1994).
- SOP-15.09, Chain of Custody for Analytical Data Packages.
- LANL (Los Alamos National Laboratory), "Environmental Restoration Project Statement of Work for Analytical Services," Revision 2, RFP Number 9-SX1-Q4257, Los Alamos National Laboratory, Los Alamos, New Mexico (July 1995).
- QP-2.2, Personnel Orientation and Training.

10.0 ATTACHMENTS

The **user** of this SOP may locate all forms associated with this procedure at <http://erinternal.lanl.gov/Quality/user/forms.asp>.

Attachment A: PEST/PCB Data-Validation Qualifier Flags, 1 page

Attachment B: PEST/PCB Data-Validation Reason Codes, 3 pages

Attachment C: Data-Validation Cover Sheet, 1 page

Attachment D: PEST/PCB Data-Validation Checklist, 1 page

[Using a token card, click here to record "self-study" training to this procedure.](#)
If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

Attachment A: PEST/PCB Data-Validation Qualifier Flags

- R The analyte is classified as “rejected.”
- J The analyte is classified as “detected,” but the reported concentration value is expected to be more uncertain than usual.
- J+ The analyte is classified as “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
The analyte is classified as “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential negative bias.
- U The analyte is classified as “not detected.”
- UJ The analyte is classified as “not detected,” with an expectation that the reported result is more uncertain than usual.

Attachment B: PEST/PCB Data-Validation Reason Codes

Code	PEST/ PCB Code	Qualifier Nondetects	Qualifier Detects	Description	Comments
3	P3	N/A	J+	The results for affected analytes are considered estimated and biased high (J+) because the associated surrogate was recovered above the upper acceptance limit.	Qualify only the detected results.
3a	P3a	N/A	J-	The results for affected analytes are considered estimated and biased low (J-) because the associated surrogate was recovered below the lower acceptance limit but greater than or equal to 10%R.	This code is used for detected analytes.
3b	P3b	N/A	J-	The results for affected analytes are considered estimated and biased low (J-) because the associated surrogate was recovered at less than 10%R.	This code is used for detected analytes.
3c	P3c	UJ	N/A	The reporting limits for affected analytes are considered estimated (UJ) because the associated surrogate was recovered below the lower acceptance limit but greater than or equal to 10%R.	This code is used for nondetected analytes.
3d	P3d	R	N/A	The reporting limits for affected analytes are considered rejected (R) because the associated surrogate was recovered at less than 10%R.	This code is used for nondetected analytes.
3e	P3e	UJ for LAL	J	The reporting limits/results for affected analytes are considered estimated (UJ)/(J) because one of the associated surrogates was above the upper acceptance limit and one was below the lower acceptance limit.	
3f	P3f	R See comments	R See comments	The required surrogate information is missing. Validation cannot proceed without this information.	Package should be returned to SMO or the information requested from the laboratory.
4	P4	N/A	UJ	The results for the affected analytes are considered not detected (UJ) because the associated sample concentration was less than 5 times the amount in the method blank.	Effective dilutions must be considered for common laboratory contaminants.
4a	P4a	N/A	J	The results for affected analytes are considered estimated (J) because the associated sample concentration was greater than 5 times the amount in the method blank.	
4b	P4b	R See comments	R See comments	The required method blank documentation is missing. Validation cannot proceed without this information.	Package should be returned to SMO or the information requested from the laboratory.
7	P7	R	J	The results for affected analytes are considered rejected (R)/estimated (J) because the associated analyte did not have a valid 5 point calibration and/or a standard at the reporting limit.	Qualify only the affected analytes.
7a	P7a	UJ	J	The results/reporting limits for affected analytes are considered estimated (J)/estimated (UJ) because the associated %RSD or %D exceeded criteria in the initial or continuing calibration standards.	Qualify only the affected analytes.
7b	P7b	N/A	J	The results for affected analytes are considered estimated (J) because the associated continuing calibration standard was not analyzed within 72 h of the initial analysis or no the pattern in the sample does not adequately match the pattern for the analyte in the standard.	Code should be used if a multicomponent analysis is reported. This code is also used if the analyte is reported but the pattern for the sample does not match the pattern for the analyte in the standard. If there is no valid initial calibration use reason code P16. If the %D is out of control use reason code P7a.

Code	PEST/ PCB Code	Qualifier Nondetects	Qualifier Detects	Description	Comments
8	P8	N/A	UJ	The reported analyte was not confirmed during the analysis of a second dissimilar column.	
8a	P8a	R See comments	R See comments	The required confirmation column analysis documentation is missing. Data may not be acceptable for use.	The package should be returned to SMO or the information requested from the laboratory.
9	P9	UJ	J-	The results/reporting limits for affected analytes are considered estimated and biased low (J-)/estimated (UJ) because the extraction holding time was exceeded.	
9a	P9a	R	R	The results for affected analytes are considered rejected (R) because the sample extraction exceeded 2 times the acceptable extraction holding time.	
9b	P9b	R	R	The results for affected analytes are rejected (R) because the analytical holding time was exceeded.	
10	P10	UJ/R See Comments	J-	The results/reporting limits for affected analytes are considered estimated and biased low (J-)/estimated (UJ)/rejected (R) because the associated breakdown criteria was exceeded.	This code is used for qualification of the DDT and Endrin only. If the compound is present qualify as (J), if the compound is not present but its breakdown products are then qualify as (R). If the compound is not present and no breakdowns are present, then qualify as (UJ).
10a	P10a	UJ	J+	The results/reporting limits for affected analytes are considered estimated (J)/estimated (UJ) because the associated breakdown criteria was exceeded.	This code is used for the breakdown products of DDT and Endrin.
10b	P10b	R See comments	R See comments	The breakdown documentation is missing. Validation cannot proceed without this information.	The package should be returned to SMO or the information requested from the laboratory.
10c	P10c	See comments	See comments	Undetected results for affected analyte are considered estimated (UJ) or rejected (R) because the laboratory diluted the sample for matrix interferences.	Qualify all the results as rejected if the laboratory cannot provide proof of clean up or matrix interferences. Qualify not detected results as estimated if the laboratory can provide evidence of clean up and/or matrix interferences not subject to acceptable clean up methods.
11	P11	R See comments	R See comments	The required retention time documentation is missing is missing. Validation cannot proceed without this information.	The package should be returned to SMO or the information requested from the laboratory.
11a	P11a	See comments	See comments	The results for the affected analytes are considered not-detected (U)/estimated (J)/rejected (R) because the associated retention times have shifted by more than 0.05 minutes from the mid-level standard of the initial calibration.	The validator must check the chromatogram profile to determine if any false positives or negatives exist. Qualify reported analytes not detected (U) if retention times are not within the initial calibration RT windows. Reject (R) analytes that are present but not reported accurately. This code is only used for the CCV.
11b	P11b	N/A	J	The results for affected analytes are considered estimated (J) because the analyte was positively confirmed but outside the retention time window.	Code is used when the CCV is in control but an individual analyte is reported as a positive result but outside the retention time window and confirmed. If the analyte is not confirmed or has the same problem on the confirmation column use reason code P8.

Code	PEST/ PCB Code	Qualifier Nondetects	Qualifier Detects	Description	Comments
12	P12	R See comments	R See comments	LCS documentation is missing. Validation cannot proceed without this information.	Package should be returned to SMO or the information requested from the laboratory.
12a	P12a	R	J-	The results/reporting limits for affected analytes should be regarded as estimated and biased low (J-)/rejected (R) because the associated LCS %R was less than 10%R.	Qualify all the analytes in that fraction (SVOC) and/or all analytes quantitated by the failing analyte.
12b	P12b	N/A	J-	The results for affected analyte are considered estimated and biased low (J-) because the associated LCS %R was less than 70%R but greater than or equal to 10%R.	Qualify all the analytes in that fraction and/or all analytes quantitated by the failing analyte. This code is for detected analytes.
12c	P12c	UJ	N/A	The reporting limits for affected analyte are considered estimated (UJ) because the associated LCS %R was less than 70%R but greater than or equal to 10%R.	Qualify all the analytes in that fraction and/or all analytes quantitated by the failing analyte. This code is for non-detected analytes.
12d	P12d	N/A	J+	The results for affected analyte are considered estimated and biased high (J+) because the associated LCS %R was greater than 130%R.	Qualify all the analytes in that fraction and/or all analytes quantitated by the failing analyte. This code is for detected analytes.
16	P16	R See comments	R See comments	The required calibration information is missing or samples were analyzed on an expired calibration. Validation cannot proceed without this information.	The package should be returned to SMO or the information requested from the laboratory. This code should also be used if the CCV's were not analyzed at the proper frequency and/or a multicomponent analyte was not analyzed during the initial calibration sequence.
19	R19	See comments	See comments	The validator identified quality deficiencies in the reported data that require qualification. Please see the Data-Validation Cover Sheet for specific details.	Apply the appropriate qualifier to identify the affect of the quality deficiency on the reported data.

Attachment C: Data-Validation Cover Sheet

☐

Rejected Data

Section I

Request Number: _____ Validation Date: _____ Lab Code: _____

Contract Laboratory Name: _____

Validator: _____ Organization: _____

Analytical Suite (check all that apply): ☐ Volatile Organics ☐ High Explosives
☐ Semivolatile Organics ☐ Inorganics
☐ Organochlorine Pesticides/Polychlorinated Biphenyls ☐ Radiochemistry

Other (describe): _____

Section II. Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. Chain-of-custody form(s)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Raw/BSS data
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. Case narrative	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. Quality control forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. Sample result form(s)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. Quantitation reports
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. Sample chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. TICs forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. Standard chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. TICs mass spectra

Identify any samples in the assigned Request Number that are missing:

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary.)

Validator's signature: _____ Date: _____

SOP-15.04, R1

Los Alamos National Laboratory
RRES-Remediation Program

Attachment D: PEST/PCB Data-Validation Checklist

Yes	No	n/a	(check one)	Assign qualifier listed below if criteria = Yes	
				Detected analyte	Undetected analyte
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. The breakdown performance check is not present.	R, P10b	R, P10b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. The breakdown performance exceeds criteria for individual or combined.	J-, P10	---a, P10
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. The Breakdown performance exceeds criteria and the associated breakdown products are present in sample.	J+, P10a	UJ, P10a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. Initial calibration is not present.	R, P16	R, P16
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. Initial calibration does not have 5 calibration points and/or low standard at or below the reporting limit.	J, P7	R, P7
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Initial calibration analyte %RSD is >20% or RSD is <0.995.	J, P7a	UJ, P7a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. The CCV is not present or analyzed at proper frequency.	R, P16	R, P16
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. Continuing calibration analyte %D is >15%.	J, P7a	UJ, P7a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. A multicomponent analyte is reported with no standard being analyzed within 72 h of the initial analysis.	J, P7b	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. The RT window information is not present.	R, P11	R, P11
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	11. RT window for individual analytes in the CCV have shifted more than 0.05 minutes from the RT window from the initial calibration.	---b, P11a	---c, P11a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	12. The method blank is not reported.	R, P4b	R, P4b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	13. The analyte detected in blank <u>and</u> sample result for analyte < 5x the amount in blank.	UJ, P4	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	14. The analyte detected in blank <u>and</u> sample result for analyte > 5x the amount in blank.	J, P4a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. The confirmation information is not present for positive results.	R, P8a	R, P8a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	16. The analyte reported was not confirmed on a second dissimilar column or detector.	UJ, P8	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	17. The analyte reported exceeds the RT window but was confirmed.	J, P11b	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	18. The multicomponent analyte is reported but does not match the pattern for the analyte in the associated standard.	J, P7b	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	19. Surrogate information is not present.	R, P3f	R, P3f
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	20. Surrogate % recovery is > the UAL.	J+, P3	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	21. Surrogate % recovery is < LAL but =10%.	J-, P3a	UJ, P3c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	22. Surrogate % recovery is < 10%.	J-, P3b	R, P3d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	23. One surrogate recovery is < LAL and one surrogate recovery is > UAL.	J, P3e	UJ, P3e
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	24. The LCS information is not present.	R, P12	R, P12
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	25. LCS % recovery is >130%.	J+, P12d	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	26. LCS % recovery is < 70% but =10%.	J-, P12b	UJ, P12c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. LCS % recovery is < 10%.	J-, P12a	R, P12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	28. The sample was extracted outside of the appropriate hold time.	J-, P9	UJ, P9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. The sample was extracted 2 times the appropriate holding time.	R, P9a	R, P9a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	30. The sample was analyzed outside the analytical holding time.	R, P9b	R, P9b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	31. The sample was diluted inappropriately.	N/A	U, P10c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	32. The other obvious data quality issues identified.	___, P19	___, P19

SOP-15.04, R1

Los Alamos National Laboratory
RRES-Remediation Program

^a Qualify primary analytes R if breakdown analytes are present, qualify primary analytes UJ if breakdown analytes are absent. See the SOP for more complete explanation. ^b Apply the U qualifier if the RT is not within the initial calibration RT window. Reject any anomalies. ^c Apply the J qualifier if the peak is within the initial calibration RT window, but the data are not reported as detected. Reject any anomalies

Attachment E: List of Acronyms and Abbreviations

CLP	contract laboratory program
COC	chain of custody
EPA	U.S. Environmental Protection Agency
EQL	estimated quantitation limit
RRES-R	risk reduction and environmental stewardship—remediation
FSF	Field Support Facility
GC/MS	gas chromatography mass spectrometry
HE	high explosives
LAL	lower acceptance limit
LANL	Los Alamos National Laboratory
LCS	(contract analytical) laboratory control sample
MDL	method detection limit
n/a	not analyzed
%D	percent difference
%R	percent recovery
%RSD	percent relative standard deviation
PEST	organochloride pesticide
PCB	polychlorinated biphenyl
QC	quality control
RL	reporting limit
RPF	Records Processing Facility
RRF	relative response factor
RT	retention time
SMO	Sample Management Office
SOP	standard operating procedure
SOW	statement of work
TIC	tentatively identified compound
UAL	upper acceptance limit